

UNCLASSIFIED

SECURITY CLASSIFICATION OF THIS PAGE (When Data Entered)

14  
18 ARO 14889.8-MS

## REPORT DOCUMENTATION PAGE

READ INSTRUCTIONS  
BEFORE COMPLETING FORM

REPORT NUMBER

2. GOVT ACCESSION NO.

3. RECIPIENT'S CATALOG NUMBER

TITLE (and Subtitle)

6 Failure Prediction in Glass,

5. TYPE OF REPORT &amp; PERIOD COVERED

Final Report  
7/1/78 to 6/30/81

6. PERFORMING ORG. REPORT NUMBER

AUTHOR(s)

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8. CONTRACT OR GRANT NUMBER(s)

DAAG29-78-G-0141

PERFORMING ORGANIZATION NAME AND ADDRESS

Rensselaer Polytechnic Institute  
Materials Engineering Department  
Troy, New York 1218110. PROGRAM ELEMENT, PROJECT, TASK  
AREA & WORK UNIT NUMBERS

DAAG29-78-G-0141

11. CONTROLLING OFFICE NAME AND ADDRESS

U. S. Army Research Office  
Post Office Box 12211  
Research Triangle Park, NC 27709

12. REPORT DATE

July 1981

13. NUMBER OF PAGES

7

14. MONITORING AGENCY NAME &amp; ADDRESS (if different from Controlling Office)

Final rept.

1 Jul 78-30 Jun 81

15. SECURITY CLASS. (of this report)

Unclassified

15a. DECLASSIFICATION/DOWNGRADING  
SCHEDULE

16. DISTRIBUTION STATEMENT (of this Report)

Approved for public release; distribution unlimited.

17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)

NA

18. SUPPLEMENTARY NOTES

The view, opinions, and/or findings contained in this report are those of the author(s) and should not be construed as an official Department of the Army position, policy, or decision, unless so designated by other documentation.

19. KEY WORDS (Continue on reverse side if necessary and identify by block number)

Glass, life prediction, fatigue, fracture

20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

The static fatigue of Pyrex borosilicate glass was measured over a wide range of stress and failure time, and the log failure time was found to be inversely proportional to the applied stress. The appearance of fracture origins was consistent with crack tip sharpening during fatigue, as predicted by the Hillig-Charles theory as modified for different failure time-stress functional relations. An explanation for the sensitivity of fatigue of soda-lime glass to surface treatment was proposed.

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FAILURE PREDICTION IN GLASS

FINAL REPORT

Robert H. Doremus

July 1981

U. S. Army Research Office

Grant DAAG29 78 G 0141  
Contract DAAG29 80 C 0140

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## Failure Predution in Glass

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### Purpose

The purpose of this work was to study the fatigue of glass both experimentally and theoretically to provide a basis for extrapolating experimental fatigue data to long times (many years). The experimental methods used were to measure delayed failure times for Pyrex borosilicate glass over a wide range of stress and time, and an examination of fracture origins in soda-lime and Pyrex glasses in the scanning electron microscope. The fatigue results were examined by statistical techniques to determine the functional dependence of failure time on stress. The theory of Hillig and Charles was modified by new stress-time functions, and an explanation of variations in fatigue of glass with different surface treatments proposed. The calculation of parameters in the Weibull distribution function was examined to determine their reliability. The results of this work are summarized in the next few paragraphs; details of the work are given in the publications listed in the bibliography.

Extensive data on the static fatigue of Pyrex borosilicate glass have been taken over a wide range of failure times and stresses. Many (50 to 100) samples were held to failure at each of a number of stresses; a total of more than 1200 samples were fractured. Since the reliability of the mean log failure time at a particular stress is proportional to  $\sigma/\sqrt{n}$ , where  $n$  is the number of samples and  $\sigma$  their standard deviation, these means were known quite accurately because of the large number of

samples, in spite of the large  $\sigma$  of about 25%. Experimental handling and conditions such as relative humidity and temperature were the same for all tests. It was found that the stress at the sample could not be calculated accurately enough from the ratio of beam arms and applied weights; it was necessary to measure the strain on the sample directly with strain gages.

The results were used to examine various equations for the dependence of failure time  $t$  on stress:

$$\log t = a - bS/S_N \quad (1)$$

$$\log t = c - n \log S/S_N \quad (2)$$

$$\log t = d + g S_N/S \quad (3)$$

The applied stress is normalized with the failure stress  $S_N$  at liquid nitrogen temperature.

A regression (least squares) analysis of the fit of eqs. 1-3 to the fatigue data showed that eq. 3 fit the data best, with eq. 1 being much inferior. This fit was confirmed in two other ways. The variation of the standard deviation  $\sigma$  with failure time is related to the functional dependence of  $\log t$  on  $S$  (eqs. 1-3).<sup>1</sup> For eq. 3 the standard deviation should increase with  $\log t$ ; this result was found for our data on Pyrex and data of others on soda-lime<sup>1,2</sup>, vitreous silica<sup>3</sup>, and another borosilicate glass.<sup>3</sup> Long-time failure experiments are an especially sensitive way to test the validity of stress failure time relations; only seven of sixteen samples at  $S/S_N = .25$  had failed after nearly two years. Equations 1 and 2 predicted early failure at this stress, leaving eq. 3 as the only one consistent with the results. These results are described in publication no. 6.

Fracture origins were observed in the scanning electron micro-

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scope for soda-lime and Pyrex glasses fractured at 25° and -196°C (publication no. 1). There was no significant difference in the failure mode at the two temperatures, confirming the Hillig-Charles<sup>4</sup> results (see below) that fatigue, which occurs at 25° but not -196°, is caused by tip sharpening and not crack lengthening.

The Hillig-Charles theory<sup>4</sup> was modified by using the relations:

$$v = A \sigma^n \quad (4)$$

and

$$v = v_\infty e^{-\alpha/\sigma} \quad (5)$$

between the rate of corrosion  $v$  and tip stress  $\sigma$  ( $A$ ,  $n$ ,  $\sigma$  and  $v_\infty$  are constants) instead of

$$v = v_0 e^{\beta\sigma} \quad (6)$$

as originally used by Hillig and Charles. The results are simpler for eqs. 4 and 5 than for 6, and confirm the Hillig-Charles conclusion that fatigue results from tip sharpening, (see publication no. 3)

A comparison of the normal and Weibull distribution functions for fracture and fatigue data suggested that the normal distribution is more convenient and fits data better, in spite of widespread use of the Weibull. When the fit of data to either distribution is not good ( $R^2 < .9$ ), the Weibull is especially inconvenient, because reliable values of the parameter  $m$  and  $S_0$  (spread and scaling) can be found only by the maximum likelihood method,<sup>5,6</sup> which requires a complicated iterative calculation. This work is being prepared for publication.

There has been no satisfactory explanation for the great sensitivity of failure times of soda-lime glass to different surface treatments such as abrasion and aging a freshly abraded surface in water. An explanation

was proposed (publication no. 4) involving different distributions of crack tip radii. Unfortunately crack tip radii have not been observed, so the explanation cannot be checked directly.

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